

Characterization of SiGe/Ge Heterostructures and Graded Layers Using Variable Angle Spectroscopic Ellipsometry

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Variable angle spectroscopic ellipsometry (VASE) has been used to characterize $\text{Si}_x\text{Ge}_{1-x}/\text{Ge}$ superlattices (SLs) grown on Ge substrates and thick $\text{Si}_x\text{Ge}_{1-x}/\text{Ge}$ heterostructures grown on Si substrates. Our VASE analysis yielded the thicknesses and alloy compositions of all layers within the optical penetration depth of the surface. In addition, strain effects were observed in the VASE results for layers under both compressive and tensile strain. Results for the SL structures were found to be in close agreement with high resolution x-ray diffraction measurements made on the same samples.

The VASE analysis has been upgraded to characterize linearly graded $\text{Si}_x\text{Ge}_{1-x}$ buffer layers. The algorithm has been used to determine the total thickness of the buffer layer along with the start and end alloy composition by breaking the total thickness into many (typically > 20) equal layers. Our ellipsometric results for 1 μm buffer layers graded in the ranges $0.7 \leq x \leq 1.0$ and $0.5 \leq x \leq 1.0$ are presented, and compare favorably with the nominal values.

INTRODUCTION

Characterization of semiconductor superlattice (SL) structures is typically done by x-ray diffraction (XRD) and transmission electron microscopy (TEM). Both XRD and TEM can be used to quantify SL ordering and periodicity. In addition, XRD can give the average value of the composition. Both techniques have limitations; TEM is destructive, XRD requires relatively thick samples ($\sim 1000 \text{ \AA}$), and neither technique can be used to quantify individual interfaces or quantum wells. Recently, variable angle spectroscopic ellipsometry (VASE) has been shown to be a powerful, non-destructive technique for the post-deposition characterization of $\text{Si}_x\text{Ge}_{1-x}/\text{Si}$ SLs and other multilayer heterostructures [1,2]. In these studies VASE has been used to determine layer thicknesses, alloy composition, oxide thickness, number of superlattice periods, and sample homogeneity. In this current work we will concentrate on Ge rich $\text{Si}_x\text{Ge}_{1-x}/\text{Ge}$ SLs and heterostructures.

Graded composition $\text{Si}_x\text{Ge}_{1-x}$ layers are used in the base of $\text{Si}_x\text{Ge}_{1-x}/\text{Si}$ heterojunction bipolar transistors to increase device speed. Graded composition $\text{Si}_x\text{Ge}_{1-x}$ layers (graded to achieve $x \approx 0.3$) are also

used as buffers to relieve strain in the growth of n-type $\text{Si}_x\text{Ge}_{1-x}/\text{Si}$ modulation doped field effect transistors (MODFET) structures. In previous VASE analysis of MODFETs, only the high energy portion of the ellipsometric spectra was used. Thus, a graded composition layer analysis was avoided, since the graded layer was buried below the optical penetration depth of the probing light at these energies. In this work a simple algorithm for the calculation of the Fresnel reflection coefficients of a linearly graded $\text{Si}_x\text{Ge}_{1-x}$ layer has been developed. This allowed a graded $\text{Si}_x\text{Ge}_{1-x}$ layer to be characterized in terms of its thickness, and Si content at the substrate and ambient surfaces.

BACKGROUND

To determine the properties of multilayer structures the measured ellipsometric angles, $\{\tan \Psi(\lambda), \cos \Delta(\lambda)\}$, must be compared to results calculated from well defined models. Linear regression analysis (LRA) is used to minimize the unbiased estimator, σ :

$$\sigma^2 = \frac{1}{n - m - 1} \sum_{i=1}^n \{ (\tan \Psi_i^e - \tan \Psi_i^c)^2 + (\cos \Delta_i^e - \cos \Delta_i^c)^2 \} \quad (1)$$

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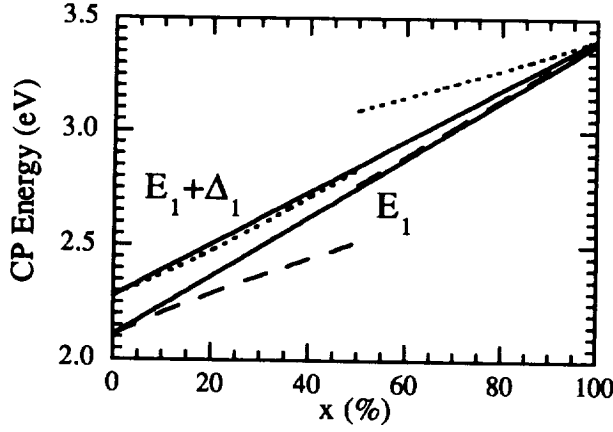


Figure 1: E_1 (long dash) and $E_1 + \Delta_1$ (short dash) critical point energies for relaxed and strained $\text{Si}_x\text{Ge}_{1-x}$ layers. The relaxed curves (Ref. 6) are shown as solid lines. The strain induced splitting (dashed lines) was calculated using Eqs. 2 and 3. For $x > 0.5$ the substrate is Si and the strain is compressive, and for $x < 0.5$ the substrate is Ge and the strain is tensile.

where n is the number of observed data points and m is the number of free parameters used in the model. The superscripts e and c refer to the experimentally observed data and the corresponding results calculated by the model, respectively. In order to construct a model, the dielectric function of all the constituent materials must be known. The dielectric function of composite layers can be determined using effective medium approximations (EMAs) [3]. The energy shift algorithm [4] is used to interpolate between $\text{Si}_x\text{Ge}_{1-x}$ dielectric functions published at discrete alloy compositions [5]. The algorithm requires a knowledge of the functional dependence of the critical point (CP) energies with the alloy composition, usually the $E_0(x)$, $E_1(x)$, and $E_2(x)$ CP energies. To improve the analysis of Ge rich $\text{Si}_x\text{Ge}_{1-x}$ materials the energy shift algorithm has been modified to include the effects of the $E_1 + \Delta_1$ CP energy [6].

Fig. 1 shows the E_1 and $E_1 + \Delta_1$ CP energies for strained and relaxed $\text{Si}_x\text{Ge}_{1-x}$ layers. The in-plane strain, ϵ , in the strained $\text{Si}_x\text{Ge}_{1-x}$ layer results from the lattice mismatch between the layer and the substrate. The shifts in the E_1 and $E_1 + \Delta_1$ CP energies for a biaxial (001) strain are given by [7]:

$$\Delta E_1 = \frac{+\Delta_1}{2} + E_H - \frac{1}{2} (\Delta_1^2 + 4E_S^2)^{1/2} \quad (2)$$

$$\Delta(E_1 + \Delta_1) = \frac{-\Delta_1}{2} + E_H + \frac{1}{2} (\Delta_1^2 + 4E_S^2)^{1/2} \quad (3)$$

where the hydrostatic shift, E_H , and uniaxial shear, E_S , are given by:

$$E_H = 2\mathcal{E}_1(1 - C_{12}/C_{11})\epsilon \quad (4)$$

$$E_S = (2/3)^{1/2} D_3^3 (1 + 2C_{12}/C_{11})\epsilon \quad (5)$$

Table I: Comparison of target sample structure with that determined by HRXRD and VASE. The period is the sum of the Ge and SiGe layer thicknesses, and $x(\text{avg})$ is the average silicon content in one period. The parameters of the VASE analysis are the oxide, Ge and SiGe layer thicknesses, and the silicon content, x , of the SiGe layer.

Sample	Source	Period (Å)	$x(\text{avg})$ (%)	$d(\text{Ge})$ (Å)	$d(\text{SiGe})$ (Å)	x (%)
HA57	target	178	6.2 ^a	128	50	22.0
HA57	HRXRD	201.1	8.0	141.8 ^b	59.3 ^b	26.6 ^b
HA57	VASE ^c	202.8	8.4 ^a	127.5 ±3.9	75.3 ±3.5	22.7 ±1.6
HA58	target	201	7.8 ^a	142	59	26.6
HA58	HRXRD	202.1	8.3	141.8 ^b	60.3 ^b	27.5 ^b
HA58	VASE ^d	204.5	8.4 ^a	126.9 ±4.7	77.6 ±4.4	22.1 ±2.0

^aCalculated from $d(\text{Ge})$, $d(\text{SiGe})$, and x .

^bEstimated from the period, $x(\text{avg})$, and shutter opening/closing times.

^c $\sigma = 0.0137$, $d(\text{oxide}) = 17.9 \text{ Å} \pm 0.4 \text{ Å}$.

^d $\sigma = 0.0167$, $d(\text{oxide}) = 25.5 \text{ Å} \pm 0.6 \text{ Å}$.

C_{ij} are the elastic stiffness constants, \mathcal{E}_1 is the hydrostatic deformation potential, and D_3^3 is an intraband deformation potential for the Λ_3 valence band for a [001] uniaxial strain. The energy shift algorithm assumes the functional dependence of the relaxed E_1 and $E_1 + \Delta_1$ CP energies shown in Fig. 1. Therefore, when modeling strained $\text{Si}_x\text{Ge}_{1-x}$ layers the strain induced shifts in the E_1 and $E_1 + \Delta_1$ will cause the value of the Si content, x , to be over- or under-estimated for a compressive or tensile strain, respectively.

SiGe/Ge SUPERLATTICE STRUCTURES ON Ge SUBSTRATES

Two fifty period $\text{Si}_x\text{Ge}_{1-x}/\text{Ge}$ SL structures, HA57 and HA58, were grown by Si molecular beam epitaxy (MBE) on Ge (100) substrates at Hughes Research Laboratories. The samples were first characterized by high resolution x-ray diffraction (HRXRD) [8], the results are shown in Table I along with the target structures. Each sample was then measured by VASE at several angles of incidence (69° , 73° , and 77°) which were chosen to increase the sensitivity of the ellipsometric angles to the sample structure parameters [9]. The measured $\{\tan \Psi, \cos \Delta\}$ spectra for sample HA57 are shown in Fig. 2 along with the spectra generated from the best fit model. Due to the strong absorption in the low wavelength region, discrepancies between the measured and calculated spectra in this region are a result of devia-

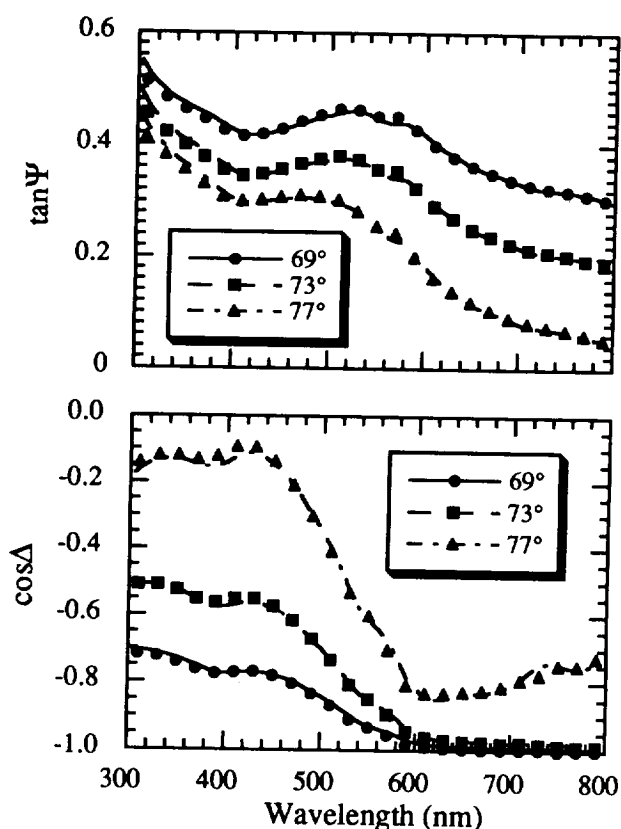


Figure 2: Comparison of measured VASE data (symbols) with that determined from the best fit model (lines) for the $\text{Si}_x\text{Ge}_{1-x}/\text{Ge}$ SL sample HA57. Sample HA58 shows similar fitting. The structure parameters found from the best fit model are shown in Table I.

tions in the model that occur near the surface. Similarly, discrepancies in the fitting in the long wavelength region are most likely due to problems in the model which occur deeper in the structure. In this case the model assumes that each period of the SL is identical. Some slight oscillations can be seen in the long wavelength region of the $\cos \Delta$ spectra which are not reproduced by the model. This indicates that the periods of the sample are not identical and that the thickness and/or $x(\text{avg})$ of each period may be fluctuating about some average value.

The agreement between the HRXRD and VASE results is quite good, especially when comparing the period and $x(\text{avg})$. There is, however, a substantial difference between the individual layer thicknesses and x . The $\text{Si}_x\text{Ge}_{1-x}$ layer is under a tensile strain as a result of being sandwiched between the thicker Ge layers. This tensile strain accounts for the lower x value obtained by VASE. The differences in the layer thicknesses may be a result of interfacial layers which are not currently accounted for in the model.

Nominal		VASE	
	Oxide	$\text{Si}_x\text{Ge}_{1-x}$ (49%)	36.5 Å
		GeO (51% ± 11%)	± 6.5 Å
200 Å	$\text{Si}_{0.3}\text{Ge}_{0.7}$	$\text{Si}_x\text{Ge}_{1-x}$	206.1 Å
		$x = 29.9\% \pm 0.8\%$	± 10.1 Å
40 Å	Ge	Ge	34.1 Å
			± 10.7 Å
1 μm	Stepped SiGe Buffer	$\text{Si}_y\text{Ge}_{1-y}$ Substrate	
		$y = 25.1\% \pm 5.2\%$	
		$\sigma = 0.0284$	
	Si Substrate		

Figure 3: Structure of sample CL141 showing the target structure parameters and those determined from the VASE analysis.

SiGe/Ge HETEROSTRUCTURES ON SI SUBSTRATES

Two $\text{Si}_x\text{Ge}_{1-x}/\text{Ge}$ heterostructures, CL141 and CL171, were grown on Si substrates at the University of California at Los Angeles. The nominal structures for sample CL141 and CL171 are shown in Fig. 3 and Fig. 4, respectively. The Si content of the stepped buffer was changed from 100% to 20% or 30% in approximate steps of 25%. Therefore, it would require six parameters to completely characterize the buffer layer. Characterization of the buffer layer is further complicated by the fact that it is buried deep in the structure. As a result sample CL141 was modeled over the range 300 nm to 550 nm to ensure that the top step of the buffer would act as the substrate. Over this range the maximum penetration depth is ~1000 Å and ~200 Å in $\text{Si}_{0.3}\text{Ge}_{0.7}$ and Ge, respectively. The oxide has been modeled as a mixture of $\text{Si}_x\text{Ge}_{1-x}$ (x same as in underlying layer) and GeO_2 using the Bruggeman EMA; this mixture simulates a surface roughness. The surface of these samples is expected to be rough due to the large lattice mismatch between the Ge layer and the underlying Si substrate (~4%). The VASE results, shown in Fig. 3, agree with in the 90% confidence limits with the nominal structure.

The second sample, CL171, was modeled over the spectral range 300 nm to 760 nm. Over this range the $\text{Si}_x\text{Ge}_{1-x}$ layer will act as the substrate due to the increased thicknesses of both the Ge and SiGe layers. The measured VASE data is shown in Fig. 5 along with spectra generated from the best fit model; the resulting structure parameters are shown in Fig. 4. Modeling the

Nominal		VASE	
	Oxide	Ge (38%)/	36.0 Å
		GeO ₂ (62% ± 9%)	± 2.6 Å
1000 Å	Ge	Si _y Ge _{1-y}	1263 Å
		y = 5.8% ± 0.3%	± 11 Å
1200 Å	Si _{0.2} Ge _{0.8}	Si _x Ge _{1-x} Substrate	
		x = 16.5% ± 1.5%	
1 μm	Stepped SiGe Buffer	σ = 0.0269	
Si Substrate			

Figure 4: Structure of sample CL171 showing the target structure parameters and those determined from the VASE analysis.

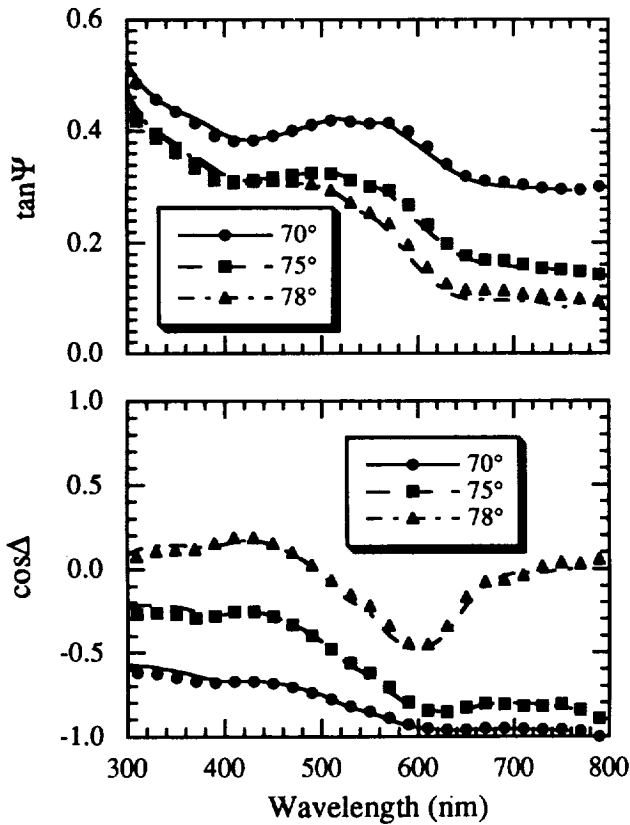


Figure 5: Comparison of measured VASE data (symbols) with that generated from the best fit model (lines) for the thick Si_xGe_{1-x}/Ge heterostructure sample CL171. The best fit model is shown in Fig. 4 along with the resulting values of the structure parameters.

Table II: Results of VASE analysis of graded Si_xGe_{1-x} layers on Si substrates. The nominal thickness of all samples is 1 μm. The samples were linearly graded from 100% Si at the substrate to x_n(nom) at the surface. The parameters of the VASE analysis are the oxide thickness, buffer layer thickness (*D*), and the Si content at the surface (x_n). The Si content at the substrate was held constant at 100%. A value of *n* = 30 was used to model all three samples.

Sample	σ	<i>d</i> (oxide) (Å)	<i>D</i> (μm)	x _n (%)	x _n (nom) (%)
HA82	0.0522	44.8 ± 0.9	1.04 ± 0.04	76.7 ± 0.6	70
HA83	0.0524	59.3 ± 1.1	1.02 ± 0.03	66.7 ± 0.6	50
HA17	0.0202	43.0 ± 0.4	0.97 ± 0.01	58.7 ± 0.3	50

Ge layer as Si_xGe_{1-x} resulted in a ~25% decrease in σ. Since the Si content of this layer should in fact be 0%, the value of 5.8% can be attributed to a compressive strain which results in an over-estimate of the Si content.

SiGe GRADED LAYERS

Three continuously, linearly graded Si_xGe_{1-x} layers were grown by Si MBE on Si (100) substrates at Hughes Research Laboratories; the nominal structures are described in Table II. Graded layers are simulated in the VASE model by breaking the layer in to *n* + 1 sub layers. The thickness of the *i*-th sub layer is:

$$d_i = \begin{cases} D/2n & i = 0, n \\ D/n & 1 \leq i < n \end{cases} \quad (6)$$

where *D* is the total layer thickness. For a linearly graded layer the Si content of the *i*-th layer is given by:

$$x_i = \frac{(n-i)x_0 + ix_n}{n} \quad (7)$$

where *x*₀ and *x*_{*n*} are the Si content at the substrate and surface, respectively. As *n* → ∞ this model will more closely approximate a continuously graded layer. However, values of *n* > 20 were found to yield nearly identical results in the VASE analysis for the graded samples used in this study.

The graded samples were measured by VASE at three angles of incidence: 70°, 75°, and 77°. Fig. 6 shows the experimental data for sample HA17 along with the ellipsometric angles generated from the best fit model. The fitting is nearly perfect in the cos Δ spectrum and in the long wavelength region of the tan Ψ spectrum. The poor fitting in the low wavelength region of the tan Ψ spectrum is most likely due to poor surface

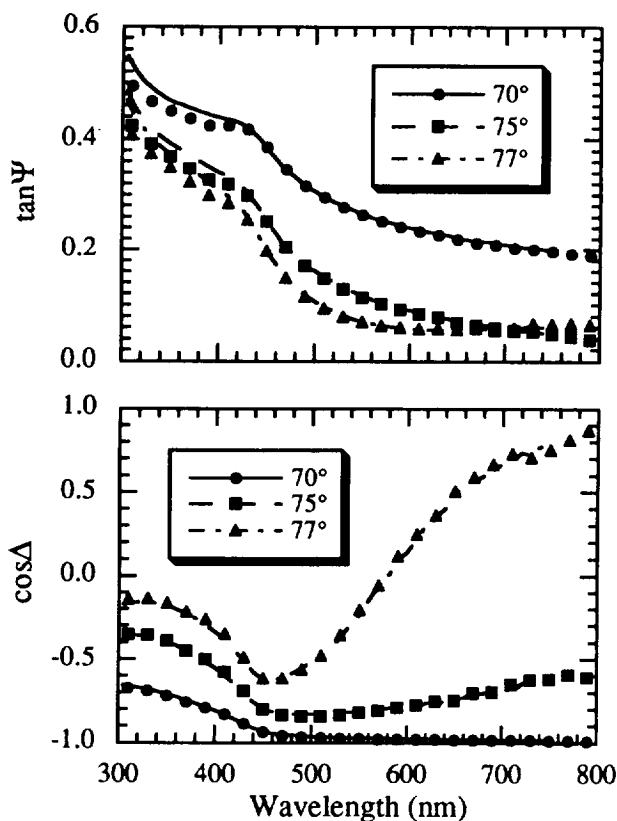


Figure 6: Comparison of measured $\{\tan \Psi, \cos \Delta\}$ spectra (symbols) for the graded $\text{Si}_x\text{Ge}_{1-x}$ sample HA17 with that determined from the best fit model (lines). The structure parameters determined from the model are shown in Table II.

quality. Because the graded layers are uncapped, the surface region of the graded layers are expected to be strained which can cause the surface to become rough. Table II summarizes the results of the VASE analysis. It can be seen from this table that the thickness values are very close to the nominal values. The values for the Si content at the surface (x_n), however, are consistently much higher than the nominal values. These discrepancies can be caused by the compressive strain in the surface region of the layer which would result in an overestimate of x . The growth log notes that the surface morphology of sample HA83 is poor when compared with sample HA82. This may be the cause of the larger discrepancy in x_n for sample HA83.

CONCLUSIONS

VASE has been used to characterize $\text{Si}_x\text{Ge}_{1-x}/\text{Ge}$ SL structures grown on Ge substrates. The values of the period and $x(\text{avg})$ determined by VASE show excellent agreement with those same values determined by HRXRD. Due to strain effects or interfacial layers not accounted for in the VASE models, the individual layer

thicknesses and x determined by the two methods do not agree as closely. Thick $\text{Si}_x\text{Ge}_{1-x}/\text{Ge}$ heterostructures grown on Si substrates have also been characterized by VASE. Results show close agreement with the nominal structure. In addition VASE analysis provides a qualitative assessment of the surface roughness and strain in the various layers of the structures. Finally, VASE has been used to characterize continuously, linearly graded $\text{Si}_x\text{Ge}_{1-x}$ layers in terms of the layer thickness and Si content at the surfaces. Results agree extremely well with the target structure, especially when strain in the surface region of these uncapped graded layers is taken in to account.

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